भारतीय मानक

Indian Standard

IS 1448 [P : 163] : 2018

पेट्रोलियम और उसके उत्पादों के लिए परीक्षण के तरीके

[पी : 163]

गैस क्रोमैटोग्राफी द्वारा इस्तेमाल के लिए पेट्रोल इंजन के तेल में
गैसोलीन मंदक का निर्धारण

Methods of Test for Petroleum and Its Products

[P : 163]

Determination of Gasoline Diluent in used Gasoline Engine
Oils by Gas Chromatography

ICS 75.100

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Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard [P : 163] was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Considerable assistance was derived from ASTM D3525 - 04 (2012) 'Standard test method for gasoline diluent in used gasoline engine oils by gas chromatography', in preparation of this standard.

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

In reporting the results of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 163]

DETERMINATION OF GASOLINE DILUENT IN USED GASOLINE ENGINE OILS BY GAS CHROMATOGRAPHY

1 SCOPE

1.1 This standard prescribes gas chromatographic techniques equipped with flame ionization detector and programmable oven to determine fuel dilution of used gasoline fuel engine oils. A sample containing a known percentage of *n*-tetra decane as primary standard is used to determine the weight percent of gasoline fuel in the sample boiling below the boiling point of the primary standard.

1.2 This test method measures and indicate the fuel dilution of the engine oil during normal operation. Fuel dilution of the crankcase oil retards the performance of the engine oil however in normal performance some fuel dilution may take place.

2 APPARATUS

2.1 Gas Chromatograph — Gas chromatograph having the following performance characteristics can be used:

2.1.1 *Detector* — Flame Ionization Detector (FID) having sufficient sensitivity to detect 1.0 percent *n*-tetra decane with a peak height of at least 40 percent of full scale on the recorder. The detector must be capable of operating continuously at a temperature equivalent to the maximum column temperature employed, and it must be connected to the column so as to avoid any cold spots. When operating at this sensitivity level, detector stability must be such that the baseline drift of not more than 1 percent per hour is obtained.

2.1.2 *Column Temperature Programmer* — Capable of temperature programme operation over a range sufficient to establish a retention time of 0.25 min (15 s) for the initial peak and to elute the internal standard totally. For determination of fuel dilution the reproducibility of the programming rate is not significant, although a retention time repeatability of 0.3 min (18 s) should be available.

2.1.3 *Sample Inlet System* — Capable of operating continuously at a temperature equivalent to the maximum column temperature employed. The sample inlet system must be connected to the chromatograph column so as to avoid any cold spot.

2.2 Recorder — Recording potentiometer with a full-scale response time of 1 s or less.

2.3 Column — Column capable of providing separations in order of boiling points under the conditions of the test.

2.4 Computer or Electronic Integrator — For measuring the retention times, eluting peak areas and accumulated area under the chromatogram.

2.5 Flow Controllers — Gas chromatograph equipped with constant-flow controllers to control drift to not more than 1 percent of full scale per hour.

2.6 Micro Syringe, automatic sampling devices, or indium encapsulation, for sample introduction.

2.7 Syringes, 1 ml, graduated in 0.01 ml, 25 µl and 1 µl.

2.8 Vials, 1 dram (3.7 ml), septum-capped.

3 REAGENTS

3.1 Liquid Phase for Columns — Any nonpolar liquid phase suitable for column operation above 300°C may be used.

3.2 Solid Support — Crushed fire brick or diatomaceous earth, in the case of packed columns. Where solid support is used, sieve size and support loading should be such as will give optimum resolution and analysis time. In general, support loading of 3 to 10 percent has been found most satisfactory.

3.3 Carrier Gas — Helium, nitrogen, or argon shall be used with the flame ionization detector.

3.4 *n*-Tetradecane of 95 percent minimum purity.

3.5 *n*-Hexadecane of 95 percent minimum purity.

3.6 *n*-Octane of 95 percent minimum purity.

4 TYPICAL GAS CHROMATOGRAPHIC CONDITIONS

Column ID, in. (mm)	0.093 (2.36)
Column length, ft (m)	2 (0.610)
Liquid phase	Dexsil 300A

Percent liquid phase	10
Support material	Chromosorb WAWB
Support mesh size	80/100
Initial column temperature, °C	30
Final column temperature,°C	255
Programming rate, °C/min	6
Carrier gas	Helium or nitrogen or argon
Carrier gas flow rate, ml/min	30
Detector	FID
Detector temperature (manifold),°C	260
Injection port temperature, °C	255
Sample size, µl	0.7
Column resolution	4.85

5 PROCEDURE

5.1 Preparation of Apparatus

5.1.1 *Column Preparation* — minimum Method used in gas chromatography that will produce a column meeting the requirements.

5.1.2 To test column resolution prepare a mixture of 1 volume percent each of C_{14} and C_{16} normal paraffins in a suitable solvent such as octane.

5.2 Chromatograph and Related Equipment — All preparations must be made in line with the manufacturer's instructions. Typical operating conditions as given in **4**.

5.2.1 The deposits formed in the flame ionization detector from combustion must be removed regularly, since they change the response characteristics of the detector.

5.2.2 If the sample inlet system is heated above 300°C, a blank run must be made after new septums are installed, to check for extraneous peaks produced by septum bleed. At the sensitivity levels employed in this method, conditioning of the septum at the operating temperature of the sample inlet system for several hours will minimize this problem. Recommended practice is to change septums at the end of the day's operation rather than at the beginning.

5.3 Preparation of Sample

Inject 0.5 ml of sample in a tared 1-dram vial using a 1-ml syringe, weigh, and record. Add 10 µl of *n*-tetradecane (C_{14}), weigh, and record. If the sample is too viscous for injection into the gas chromatograph at room temperature, add 0.5 ml of a suitable solvent to the vial. Use of *n*-hexadecane has been found to be acceptable as a solvent. Stopper the vial with a septum cap and shake for at least 2 min.

5.4 Method

5.4.1 Inject the desired volume of sample. Sample size must be taken very carefully. With hydrogen flame ionization detectors, the usual sample size ranges from 0.2 to 1.0 µl. The syringe needle must remain in the injection port manifold for 5 to 10 s after injection to avoid any sample fractionation in the needle.

5.4.2 Immediately start programming the column temperature upward at the rate given in **4**. Turn on the recorder chart drive and integrator immediately after injection of the sample. Set the attenuation to allow maximum peak height of the area prior to and including the *n*-tetra decane reference peak without overloading the method of measurement.

5.4.3 Area accumulation and recording may be discontinued after elution of the *n*-tetra decane. This method is not intended to define the nature of the lubricating oil peak. Removal of non-eluted oil from the system prior to subsequent operation may be achieved by techniques such as back flush or increased system temperatures.

6 CALCULATION

6.1 Record the cumulative area under the chromatogram from time zero to the start of the *n*-tetra decane peak. Record the area under the *n*-tetra decane peak.

6.2 Calculate the weight percent (mass percent) of fuel dilution in the sample as follows:

$$F = \frac{A_1 \times W_1 \times 100}{X_1 \times A_2}$$

where

F = fuel dilution, mass percent,

A_1 = area counts under the chromatogram before the *n*-tetra decane peak,

A_2 = area counts under the *n*-tetra decane peak on the chromatogram,

W_1 = mass of *n*-tetra decane, and

X_1 = mass of sample.

6.3 Report the result to the nearest 0.01 mass percent of the fuel.

7 PRECISION AND BIAS

7.1 The following criteria should be used for judging the acceptability of results.

7.1.1 *Repeatability*

The difference between successive test results, obtained by the same operator should be considered suspect if it exceeds by 0.28 mass percent in more than one case out of twenty.

7.1.2 *Reproducibility*

The difference between two, single and independent results, obtained by different operators should be considered suspect if it exceeds 1.64 mass percent in more than one case out of twenty.

7.2 Bias

No estimate of the bias of this test method is possible because of the empirical nature of this test method.

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Amendments Issued Since Publication

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